rapport à la configuration qu'il possède lorsqu'il a fixé un proton dans le chlorhydrate d'histidine monohydraté.

Ce travail a été rendu possible grâce à l'utilisation de l'ordinateur IBM 7044 du Centre de Calcul Numérique de la Faculté des Sciences de Toulouse. Nous remercions très vivement toutes les personnes de ce centre qui nous ont permis de toujours exploiter dans les meilleurs délais les différents programmes utilisés.

Références

ANDREWS, A. C. & LYONS, T. D. (1957). *Science*, 126, 561.
ANDREWS, A. C., LYONS, T. D. & O'BRIEN, T. D. (1962). *J. Chem. Soc.* p. 1776.
ANDREWS, A. C. & ROMARY, J. K. (1964). *J. Chem. Soc.* p. 405.
BLOUNT, J. F., FRASER, K. A., FREEMAN, H. C., SZYMANSKI, J. T. & WANG, C.-H. (1967). *Acta Cryst.* 22, 396.
BOYKO, E. R., HALL, D., KINLOCH, M. E. & WATERS, T. N. (1966). *Acta Cryst.* 21, 614.
BROWN, D. S., LEE, J. D. & MELSOM, B. G. (1968). *Acta Cryst.* B24, 730.
BROWN, D. S., LEE, J. D., MELSOM, B. G. A., HATHAWAY, B. J., PROCTER, I. M. & TOMLINSON, A. A. G. (1967). *Chem. Comm.* p. 369.
BUSING, W. R. & LEVY, H. A. (1962). *A Fortran Crystallographic Least-Squares Program.* ORNL-TM-305, Oak Ridge National Laboratory, Tennessee.
BUSING, W. R. & LEVY, M. A. (1964). ORNL-TM 306. *A Fortran Crystallographic Function and Error Program.* Oak Ridge National Laboratory, Tennessee.
CRUICKSHANK, D. W. J. (1965). *Computing Methods in Crystallography*, p. 107. 1st ed. Oxford: Pergamon Press.
DIJKSTRA, A. (1966). *Acta Cryst.* 20, 588.
DONOHUE, J. & CARON, A. (1964). *Acta Cryst.* 17, 1178.
EVERTSSON, B. (1969). *Acta Cryst.* B25, 30.

FREEMAN, H. C. (1966). *Proc. Symp. Biochem. Copper*, p. 77–113. New York: Harriman, 1965.
FREEMAN, H. C. & SMITH, J. E. W. (1966). *Acta Cryst.* 20, 153.
GURD, F. R. N. & WILCOX, P. E. (1956). *Advanc. Protein Chem.* 11, 311.
HARDING, M. M. & COLE, S. J. (1963). *Acta Cryst.* 16, 643.
HATEM, S. (1958). *C. R. Acad. Sci. Paris*, 246, 2433.
HATEM, S. (1960). *Helv. Chim. Acta*, 175, 1431.
HATEM, S. (1960). *Chimia*, 14, 130.
International Tables for X-ray Crystallography (1962). Vol. III, p. 202. Birmingham: Kynoch Press.
JAIN, P. C. & LINGAFELTER, E. C. (1967). *J. Amer. Chem. Soc.* 89, 724.
KRETSINGER, R. H. & COTTON, F. A. (1963). *Acta Cryst.* 16, 651.
LUNDBERG, B. K. S. (1966). *Acta Cryst.* 21, 901.
MADAULE-AUBRY, F. & BROWN, G. M. (1968a). *Acta Cryst.* B24, 745.
MADAULE-AUBRY, F. & BROWN, G. M. (1968b). *Acta Cryst.* B24, 754.
MARSH, R. & DONOHUE, J. (1967). *Advanc. Protein Chem.* 22, 235.
MICKEL, B. L. & ANDREWS, A. C. (1955). *J. Amer. Chem. Soc.* 77, 5291.
NORTIA, T. (1959). *Suomen Kemi.* B32, 245.
PAJUNEN, A. (1967). *Suomen Kemi.* B40, 32.
PANOTTONI, C., BOMBIERI, G. & GRAZIANI, R. (1967). *Acta Cryst.* 23, 537.
RUST, P. MURRAY & WRIGHT, J. D. (1968). *J. Chem. Soc.* A2, 247.
SUJIHARA, A., ASHIDA, T., SASADA, Y. & KAKUDO, M. (1968). *Acta Cryst.* B24, 203.
WILLIAMS, D. E. & RUNDLE, R. E. (1964). *J. Amer. Chem. Soc.* 86, 1660.
WILLIAMS, R. M. & WALLWORK, S. C. (1967). *Acta Cryst.* 23, 448.
ZAREMBOWITCH, Z. (1963). *C. R. Acad. Sci. Paris*, 256, 1290.
ZAREMBOWITCH, Z. (1965). *Communication personnelle.*
ZAREMBOWITCH, Z. (1966). *J. Chim. Phys.* 63, 445.

The Crystal Structure of *trans*-Bicyclo[4.2.0]octyl 1-3,5-Dinitrobenzoate

BY B. L. BARNETT AND RAYMOND E. DAVIS

Department of Chemistry, The University of Texas at Austin, Austin, Texas 78712, *U.S.A.*

The structure of *trans*-bicyclo[4.2.0]octyl 1-3,5-dinitrobenzoate has been studied by single-crystal X-ray diffraction techniques. This compound ($C_{15}H_{16}N_2O_6$) crystallizes in the monoclinic space group $P2_1/c$, with $a = 9\cdot606$, $b = 6\cdot276$, $c = 25\cdot276$ Å, $\beta = 92\cdot02°$. The structure was determined by the symbolic addition procedure and was refined by block-diagonal least-squares methods to a final R value of 0·052, including hydrogen atoms. The cyclobutane ring is non-planar with a dihedral angle of 147°. The C-C bond distances average 1·544 Å for the cyclobutane ring. The six-membered ring, which is in a slightly flattened chair conformation, accommodates the strain of the bicyclic system.

Introduction

Early investigations of bicyclic systems centered around the interest in the information these studies gave re-garding reaction mechanisms and reaction intermediates. These studies involved investigating reaction rates of various derivatives of bicyclic systems in solvolysis reactions. In addition to this information,

data obtained in these investigations are now being used to determine additional properties of bicyclic systems, such as ring strain. Kinetic (solvolysis) studies are presently being carried out on [4.2.0]-bicyclooctyl systems (Wiberg & Hess, 1967; Wiberg, 1967) and some studies have already been made of [3.2.0]bicycloheptyl systems (Meinwald, Anderson & Tufariello, 1967; Wiberg, 1967). To make detailed calculations of strain energy minimization in cyclic systems, structural and thermodynamic data are also necessary.

The present investigation of the crystal structure of *trans*-bicyclo[4.2.0]octyl 1-3,5-dinitrobenzoate was undertaken as part of a study of cyclobutane rings fused with other rings in both the *cis* and *trans* configurations. Perhaps an accumulation of structural data will contribute to a knowledge of the ring strain in fused cyclobutane systems, and help to explain the high reactivity of cyclobutane derivatives in solvolysis reactions.

Experimental

A sample of *trans*-bicyclo[4.2.0]octyl 1-3,5-dinitrobenzoate [hereinafter referred to as *trans*(4.2.0)] was kindly supplied by Professor K. Wiberg of Yale University. Long rectangular colorless crystals suitable for X-ray studies were obtained by recrystallization from aqueous acetone. All X-ray studies were carried out using a General Electric XRD-5 diffractometer equipped with single-crystal orienter, with Cu $K\alpha$ radiation, and at ambient room temperature ($\sim 21\,^\circ$C). The crystal used was a rectangular prism of approximately square cross-section ($0.158 \times 0.158 \times 0.515$ mm) mounted with the long dimension [010] parallel to the φ axis of the goniometer.

Plots of the intensity-weighted reciprocal lattice were used to determine crystal symmetry. Cell constants were derived from least-squares refinement using 42 independent 2θ measurements. The density was determined by flotation in aqueous silver nitrate.

$C_{15}H_{16}N_2O_6$, $M = 320.31$

$a = 9.606$ (1), $b = 6.276$ (1), $c = 25.276$ (1) Å,
$\beta = 92.02$ (1)$^\circ$

Systematic absences: $h0l, l = 2n + 1$; $0k0, k = 2n + 1$

Space group: $P2_1/c$, (No. 14)

$Z = 4$; $F(000) = 672$; $V = 1523.1$ Å^3;
$\mu = 9.4$ cm^{-1} (Cu $K\alpha$)

$D_{exp} = 1.396$, $D_{calc} = 1.401$ g.cm^{-3}

λ(Cu $K\alpha_{mean} = 1.54178$, Cu $K\alpha_1 = 1.54050$,
Cu $K\alpha_2 = 1.54433$ Å)

Three-dimensional single-crystal intensity data were collected by the stationary-crystal stationary-counter method to the limit $2\theta = 140^\circ$ (Cu $K\alpha_1$), using a balanced nickel–cobalt filter pair. Of the 2889 independent reflections measured, 2225 were taken to have observable intensity. The largest deviation from average

intensities for six periodically monitored reflections was 1.7%, indicating that the crystal had not suffered any radiation damage during the ten days of data collection. The usual Lorentz and polarization factors were applied, as well as a correction for α_1–α_2 splitting (Tulinsky, Worthington & Pignataro, 1959). An absorption correction based on the observed crystal shape was applied (Williams, 1966). The calculated values of the absorption correction factors for the data ranged between 1.14 and 1.26. Standard deviations in observed structure amplitudes, $\sigma(|F_o|)$, based on counting statistics, were calculated according to Evans (1961).

Determination and refinement of the structure

Phases for the structure solution were obtained by the symbolic addition procedure (Karle & Karle, 1966), after conversion of the data to normalized structure factor magnitudes $|E|$ by use of a K curve. The signs of 272 reflections (corresponding to 12 reflections per atom) were deduced in terms of one symbolic sign a, no phase indication being accepted at a probability lower than 0.999. No clear indication could be obtained as to which sign was represented by a. Two E maps were computed – one with each of the possible signs for a – but both maps contained sheets of six-membered rings, and the expected structure was not evident in either map. The map calculated with a positive was chosen for further inspection, on the basis that it contained a less extreme variation of peak heights above background. A total of 24 peaks, including twelve thought to represent the dinitrophenyl group, were placed into a block-diagonal least-squares calculation. After three cycles, two of these atoms were found to have very high isotropic temperature factors, and were not included in the structure factor and electron density calculations which followed. The expected structure was still not evident since the bicyclic system could not yet be recognized. Three additional cycles of least-squares refinement, followed by electron density and difference density calculations, resulted in three more atoms being rejected, but also yielded four new positions which completed the structure.

Least-squares refinement of positions and isotropic temperature factors gave an R value of 0.126 after six cycles. A difference density map calculated at this point contained peaks representing all sixteen hydrogen atoms, ranging in height from 0.5 to 0.7 e.Å^{-3}. An additional six cycles of least-squares calculations, varying overall scale factor, all positional parameters, hydrogen isotropic temperature factors and heavy atom anisotropic factors, served to complete the refinement. Shifts in the final cycle were less than one estimated standard deviation for all parameters. A final difference density map contained no features other than a randomly fluctuating background below 0.25 e.Å^{-3}. Weights equal to $1/\sigma(|F_o|)^2$ were used in the least-squares calculations, which were carried out with the block-diagonal approxi-

mation including only the observed reflections. Atomic scattering factors used were those of Stewart, Davidson & Simpson (1965) for hydrogen and of Cromer & Waber (1965) for carbon, nitrogen and oxygen.

Calculations were carried out on a Control Data 6600 computer. Structure-factor and least-squares calculations were done using a local modification of a program by Kartha (1964). All Fourier computations were carried out using the Argonne National Labor-

atory program B-149 (Gvildys, 1965). Application of the symbolic addition method was facilitated by use of the programs *SORTE*66 (Bednowitz, 1966) and *EFACT* (Bednowitz, 1966). Various other data-processing programs of local origin were used.

Final atomic parameters are given in Tables 1, 2, and 3. The estimated standard deviations were calculated from the inverse matrix of the final least-squares cycle. The final R value for these parameters is 0·052 for the observed reflections only, and is 0·073
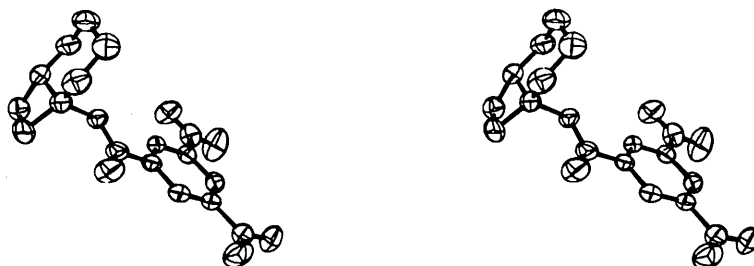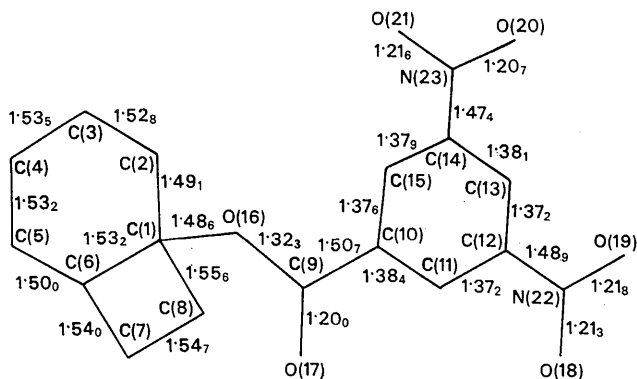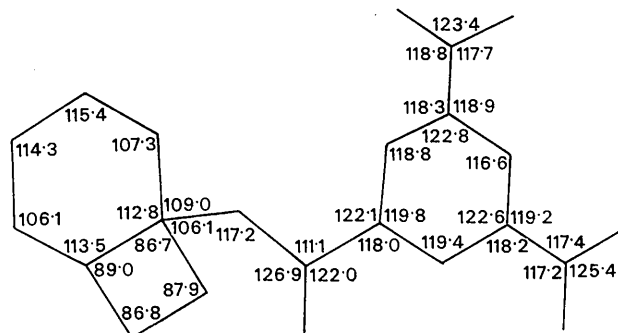

Fig. 1. Stereoscopic view of *trans*-bicyclo[4.2.0]octyl 1-3,5-dinitrobenzoate.

(*a*)

(*b*)

Fig. 2. (*a*) Bond lengths (Å). Estimated standard deviations lie in the range 0·003–0·004 Å. (*b*) Bond angles (°). Estimated standard deviations are 0·2°.

when all data are included. Observed and calculated structure factor magnitudes from the final structure factor calculation are shown in Table 4.

Table 1. *Fractional atomic coordinates (not including hydrogen atoms)*

The estimated standard deviation in parentheses refers to the last decimal position.

	x	y	z
C(1)	0·2960 (2)	0·8846 (4)	0·0934 (1)
C(2)	0·2533 (3)	0·6837 (4)	0·0660 (1)
C(3)	0·1568 (3)	0·7452 (5)	0·0191 (1)
C(4)	0·2115 (3)	0·9218 (5)	−0·0167 (1)
C(5)	0·2614 (3)	1·1222 (4)	0·0129 (1)
C(6)	0·3602 (2)	1·0470 (4)	0·0562 (1)
C(7)	0·4312 (3)	1·1558 (5)	0·1046 (1)
C(8)	0·4173 (3)	0·9400 (5)	0·1335 (1)
C(9)	0·1107 (2)	0·9035 (4)	0·1544 (1)
C(10)	−0·0160 (2)	1·0276 (4)	0·1696 (1)
C(11)	−0·1010 (2)	0·9394 (4)	0·2069 (1)
C(12)	−0·2219 (2)	1·0416 (4)	0·2191 (1)
C(13)	−0·2616 (2)	1·2317 (4)	0·1964 (1)
C(14)	−0·1730 (2)	1·3177 (4)	0·1602 (1)
C(15)	−0·0512 (2)	1·2194 (4)	0·1465 (1)
O(16)	0·1703 (1)	0·9915 (3)	0·1134 (1)
O(17)	0·1469 (2)	0·7444 (3)	0·1773 (1)
O(18)	−0·2723 (2)	0·7763 (3)	0·2788 (1)
O(19)	−0·4246 (2)	1·0258 (3)	0·2659 (1)
O(20)	−0·3165 (2)	1·6073 (3)	0·1473 (1)
O(21)	−0·1342 (2)	1·5940 (3)	0·1017 (1)
N(22)	−0·3138 (2)	0·9396 (4)	0·2579 (1)
N(23)	−0·2112 (2)	1·5204 (3)	0·1342 (1)

A comparison of the signs from the symbolic addition method with those from the final structure factor calculation showed that only one of the 272 signs used in the E map with *a* positive had been incorrect, and

that this discrepancy had been due to a clerical error. A tabulation of the major peaks of this E map revealed that the highest fifteen peaks in the map occurred at atomic positions, eleven of the next fifteen were spurious, and that nineteen false peaks appeared in this map with heights as large or larger than the smallest true atomic peak. Initial interpretation of this map was further complicated by the tendency of many spurious peaks to occur in positions completing planar six-membered rings in regions of the dinitrobenzoate group where four of the six members of the ring were at atomic sites and the additional two were spurious peaks. This occurrence of many spurious peaks in structures consisting of largely planar regions has been noticed by other workers applying direct methods, and probably could have been reduced by preliminary sign determination for a larger ratio of reflections to atoms. It may also be noted that the last six atoms found, and the six atomic peaks appearing in the E map with the lowest heights, were those of the six atoms in the bicyclic system which are furthest from the dinitrobenzoate plane.

Discussion of the structure

The structure of *trans*(4.2.0) is illustrated in the stereoscopic drawings (Johnson, 1965) of Fig. 1, which show the 50 per cent equiprobability ellipsoids derived from the anisotropic thermal parameters. Bond distances and angles are shown in Fig. 2 and also appear in Tables 5 and 6 where the standard deviations shown are those calculated from the previously estimated deviations in final atomic coordinates.

Table 2. *Anisotropic thermal parameters*

The temperature factor expression used was

$$\exp\left[-(h^2\beta_{11} + k^2\beta_{22} + l^2\beta_{33} + hk\beta_{12} + hl\beta_{13} + kl\beta_{23})\right].$$

The estimated standard deviation in parentheses refers to the last decimal position.

	β_{11}	β_{22}	β_{33}	β_{12}	β_{13}	β_{23}
C(1)	0·0103 (3)	0·0313 (10)	0·0015 (0)	0·0087 (5)	0·0003 (1)	0·0004 (2)
C(2)	0·0164 (4)	0·0274 (10)	0·0022 (1)	0·0076 (5)	0·0013 (1)	−0·0001 (2)
C(3)	0·0157 (4)	0·0352 (11)	0·0024 (1)	−0·0002 (6)	0·0004 (1)	−0·0035 (2)
C(4)	0·0171 (4)	0·0454 (13)	0·0015 (1)	0·0046 (6)	−0·0000 (1)	−0·0016 (2)
C(5)	0·0150 (4)	0·0338 (10)	0·0014 (0)	0·0014 (5)	0·0008 (1)	0·0004 (2)
C(6)	0·0095 (3)	0·0372 (11)	0·0015 (0)	0·0031 (5)	0·0006 (1)	−0·0008 (2)
C(7)	0·0110 (4)	0·0531 (14)	0·0021 (1)	−0·0028 (6)	0·0002 (1)	−0·0018 (2)
C(8)	0·0112 (4)	0·0577 (14)	0·0019 (1)	0·0128 (6)	−0·0005 (1)	0·0001 (2)
C(9)	0·0110 (3)	0·0279 (9)	0·0012 (0)	0·0044 (5)	−0·0000 (1)	0·0010 (2)
C(10)	0·0091 (3)	0·0232 (8)	0·0010 (0)	0·0015 (4)	−0·0001 (1)	0·0002 (1)
C(11)	0·0109 (3)	0·0236 (8)	0·0011 (0)	0·0001 (4)	−0·0002 (1)	0·0002 (2)
C(12)	0·0091 (3)	0·0287 (9)	0·0011 (0)	−0·0023 (4)	0·0001 (1)	−0·0003 (2)
C(13)	0·0081 (3)	0·0287 (9)	0·0014 (0)	0·0004 (4)	0·0001 (1)	−0·0018 (2)
C(14)	0·0088 (3)	0·0207 (8)	0·0013 (0)	0·0015 (4)	−0·0001 (1)	−0·0005 (2)
C(15)	0·0084 (3)	0·0242 (8)	0·0011 (0)	−0·0004 (4)	−0·0001 (1)	0·0001 (2)
O(16)	0·0092 (2)	0·0263 (6)	0·0012 (0)	0·0054 (3)	0·0006 (1)	0·0007 (1)
O(17)	0·0170 (3)	0·0394 (7)	0·0020 (0)	0·0123 (4)	0·0013 (1)	0·0042 (1)
O(18)	0·0181 (3)	0·0502 (9)	0·0028 (0)	−0·0012 (5)	0·0016 (1)	0·0054 (2)
O(19)	0·0111 (2)	0·0540 (9)	0·0026 (0)	−0·0026 (4)	0·0021 (1)	−0·0002 (2)
O(20)	0·0149 (3)	0·0396 (8)	0·0053 (1)	0·0133 (4)	0·0026 (1)	0·0032 (2)
O(21)	0·0193 (3)	0·0330 (7)	0·0026 (0)	0·0061 (4)	0·0017 (1)	0·0031 (1)
N(22)	0·0116 (3)	0·0412 (9)	0·0015 (0)	−0·0058 (4)	0·0004 (1)	−0·0005 (2)
N(23)	0·0118 (3)	0·0238 (7)	0·0022 (0)	0·0035 (4)	−0·0002 (1)	−0·0001 (2)

Table 3. *Fractional coordinates and isotropic temperature factors for the hydrogen atoms*

The estimated standard deviations in parentheses refer to the last decimal position.

	x	y	z	B
H(24)	0·2111 (20)	0·5824 (32)	0·0905 (8)	4·9 (6) Å²
H(25)	0·3389 (20)	0·6270 (33)	0·0544 (7)	5·0 (6)
H(26)	0·0637 (21)	0·7893 (35)	0·0298 (8)	5·7 (6)
H(27)	0·1282 (23)	0·6233 (37)	−0·0044 (9)	6·7 (7)
H(28)	0·1355 (22)	0·9563 (36)	−0·0446 (8)	6·5 (7)
H(29)	0·2847 (21)	0·8605 (34)	−0·0363 (8)	5·6 (6)
H(30)	0·1852 (20)	1·2033 (33)	0·0275 (8)	4·7 (6)
H(31)	0·3137 (20)	1·2260 (34)	−0·0102 (8)	5·3 (6)
H(32)	0·4338 (18)	0·9584 (31)	0·0382 (7)	3·8 (5)
H(33)	0·3690 (22)	1·2711 (38)	0·1213 (8)	6·5 (7)
H(34)	0·5291 (23)	1·2207 (39)	0·1029 (9)	7·5 (7)
H(35)	0·4933 (22)	0·8478 (36)	0·1283 (8)	6·7 (7)
H(36)	0·4025 (20)	0·9321 (34)	0·1723 (8)	5·6 (6)
H(37)	−0·0856 (18)	0·8007 (30)	0·2207 (7)	3·6 (5)
H(38)	-0·3339 (21)	1·3028 (36)	0·2084 (8)	6·3 (7)
H(39)	0·0108 (17)	1·2829 (30)	0·1232 (7)	3·0 (5)

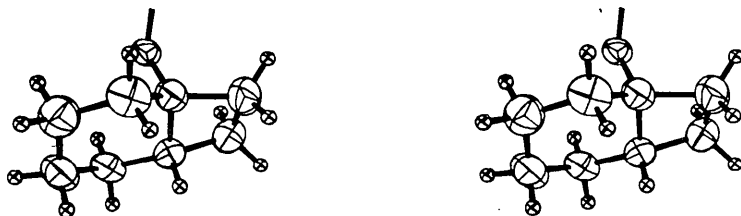Fig. 3. Stereoscopic view of the bicyclic system including the ester oxygen atom.
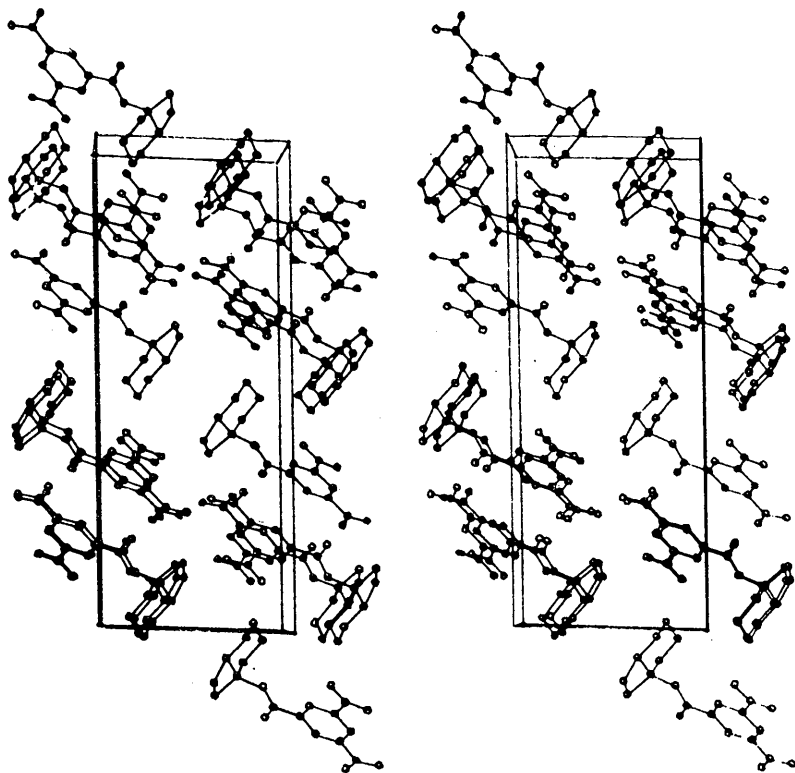

Fig. 4. Stereoscopic packing diagram. One unit-cell is outlined.

Table 4. *Observed and calculated structure factor amplitudes*

The columns headed FOBS and FCAL contain, respectively, $10|F_o|$ and $10|F_c|$.

H	K	FOBS	FCAL
••• L = -30 •••			
1	0	22	13
1	1	13	0•
2	0	48	46
2	1	30	24
3	0	27	24
••• L = -29 •••			
1	1	14	1•
1	2	13	7•
2	1	118	121
2	2	51	55
3	1	40	37
4	1	27	22
••• L = -28 •••			
1	0	50	45
1	1	31	18
1	2	29	30
1	3	21	16
2	0	170	165
2	1	34	31
2	2	43	43
2	3	44	47
3	0	15	1•
3	1	14	20•
3	2	13	5•
4	0	14	22•
4	1	61	59
4	2	21	16
5	0	22	29
5	1	12	4•
••• L = -27 •••			
1	1	47	48
1	2	35	26
1	3	59	56
2	1	16	18•
2	2	37	39
2	3	92	88
3	1	110	110
3	2	84	80
3	3	13	19•
4	1	24	15
4	2	38	31
5	1	23	16
5	2	29	25
••• L = -26 •••			
1	0	17	4•
1	1	24	21
1	2	76	72
1	3	29	27
1	4	51	59
2	0	17	27•
2	1	67	65
2	2	36	35
2	3	70	61
3	0	47	55
3	1	27	22
3	2	150	153
3	3	111	103
4	0	42	46
4	1	16	18•
4	2	96	97
4	3	13	12•
5	0	15	15•
5	1	34	29
5	2	13	7•
6	0	13	6•
6	1	31	33
••• L = -25 •••			
1	1	62	57
1	2	17	3•
1	3	16	12•
1	4	13	13•
2	1	73	75
2	2	68	71
2	3	16	17•
2	4	13	18•
3	1	93	95
3	2	17	16•
3	3	15	19•
3	4	34	29
4	1	48	42
4	2	56	47
4	3	51	51
5	1	16	4•
5	2	38	35
5	3	24	6
6	1	14	17•
6	2	13	1•
••• L = -24 •••			
1	0	34	35
1	1	34	24
1	2	103	101
1	3	67	67
1	4	27	19
2	0	37	32
2	1	41	33
2	2	70	68
2	3	30	25
2	4	28	27
3	0	18	16•
3	1	27	14
3	2	112	107
3	3	44	42
3	4	35	31
4	0	79	79
4	1	17	9•
4	2	120	120
4	3	15	6•
4	4	26	22
5	0	26	5
5	1	36	33
5	2	32	26
5	3	33	33
6	0	15	0•
6	1	78	76
6	2	14	11•
6	3	12	1•
7	0	44	43
7	1	42	37
••• L = -23 •••			
1	1	18	20•
1	2	55	48
1	3	37	31
1	4	16	17•
1	5	32	25
2	1	18	10•
2	2	95	100
2	3	88	83
2	4	46	44
3	1	106	112
3	2	90	84
3	3	107	104
3	4	15	4•
4	1	147	156
4	2	98	82
4	3	69	67
4	4	58	51
5	1	17	4•
5	2	63	59
5	3	31	26
5	4	12	2•
6	1	41	47
6	2	15	6•
6	3	13	3•
7	1	14	6•
7	2	13	21•
••• L = -22 •••			
1	0	52	46
1	1	46	51
1	2	56	59
1	3	31	32
1	4	16	16•
1	5	13	15•
2	0	51	58
2	1	28	23
2	2	33	14
2	3	117	110
2	4	16	11•
2	5	13	3•
3	0	101	107
3	1	86	90
3	2	18	17•
3	3	18	5•
3	4	16	5•
3	5	56	53
4	0	34	32
4	1	55	55
4	2	126	118
4	3	34	28
4	4	37	32
5	0	30	26
5	1	18	3•
5	2	113	111
5	3	50	52
5	4	45	49
6	0	43	44
6	1	57	57
6	2	16	11•
6	3	35	24
7	0	16	4•
7	1	15	9•
7	2	14	1•
7	3	33	32
8	0	58	50
8	1	13	15•
••• L = -21 •••			
1	1	18	12•
1	2	18	6•
1	3	95	91
1	4	74	76
1	5	15	10•
2	1	54	60
2	2	18	1•
2	3	40	38
2	4	48	47
2	5	14	18•
3	1	98	97
3	2	52	51
3	3	18	13•
3	4	16	2•
3	5	13	5•
4	1	50	55
4	2	123	121
4	3	40	28
4	4	16	14•
4	5	34	39
5	1	128	127
5	2	18	5•
5	3	47	44
5	4	14	15•
6	1	36	37
6	2	102	101
6	3	42	35
6	4	12	15•
7	1	16	8•
7	2	42	33
7	3	34	27
8	1	50	51
8	2	34	36
••• L = -20 •••			
1	0	27	12
1	1	67	65
1	2	18	19•
1	3	133	136
1	4	28	17
1	5	63	56
2	0	84	88
2	1	72	75
2	2	38	29
2	3	18	8•
2	4	83	81
2	5	15	13•
3	0	18	2•
3	1	77	101
3	2	54	54
3	3	44	48
3	4	17	17•
3	5	31	22
4	0	100	104
4	1	140	138
4	2	215	217
4	3	49	44
4	4	16	9•
4	5	42	33
5	0	177	186
5	1	181	182
5	2	264	275
5	3	17	1•
5	4	43	48
6	0	18	7•
6	1	37	41
6	2	100	95
6	3	83	80
6	4	13	13•
7	0	59	55
7	1	17	9•
7	2	78	80
7	3	14	6•
8	0	26	12
8	1	15	9•
8	2	31	27
9	0	74	75
9	1	12	5•
••• L = -19 •••			
1	1	17	32•
1	2	18	26•
1	3	80	81
1	4	18	4•
1	5	27	23
2	1	25	23
2	2	18	20•
2	3	110	100
2	4	58	63
2	5	34	32
3	1	42	33
3	2	18	1•
3	3	58	59
3	4	73	64
3	5	15	9•
4	1	30	30
4	2	102	100
4	3	18	25•
4	4	35	32
4	5	14	1•
5	1	78	75
5	2	18	0•
5	3	44	40
5	4	16	1•
5	5	13	2•
6	1	45	39
6	2	96	102
6	3	17	4•
6	4	26	16
7	1	17	3•
7	2	32	28
7	3	15	7•
7	4	12	4•
8	1	16	2•
8	2	34	34
8	3	12	15•
9	1	66	59
••• L = -18 •••			
1	0	378	386
1	1	105	102
1	2	59	54
1	3	231	241
1	4	72	78
1	5	17	9•
1	6	13	9•
2	0	89	87
2	1	31	26
2	2	90	90
2	3	58	55
2	4	34	27
2	5	40	37
2	6	25	23
3	0	25	26
3	1	106	104
3	2	85	83
3	3	112	116
3	4	80	83
3	5	16	21•
4	0	100	99
4	1	57	53
4	2	18	8•
4	3	18	3•
4	4	18	14•
4	5	35	25
5	0	86	90
5	1	215	213
5	2	94	94
5	3	40	39
5	4	17	4•
5	5	25	22
6	0	30	23
6	1	18	5•
6	2	18	1•
6	3	17	25•
6	4	34	38
7	0	44	47
7	1	33	31
7	2	51	48
7	3	49	45
7	4	40	41
8	0	139	138
8	1	29	17
8	2	15	2•
8	3	35	22
9	0	14	22•
9	1	27	26
9	2	80	83
••• L = -17 •••			
1	1	16	2•
1	2	66	71
1	3	182	183
1	4	33	23
1	5	77	64
1	6	14	1•
2	1	99	98
2	2	79	83
2	3	18	5•
2	4	99	97
2	5	17	9•
2	6	13	3•
3	1	97	90
3	2	18	9•
3	3	18	15•
3	4	54	47
3	5	28	15
3	6	28	20
4	1	18	15•
4	2	51	55
4	3	51	53
4	4	49	43
4	5	16	13•
5	1	289	292
5	2	103	91
5	3	87	84
5	4	55	55
5	5	14	5•
6	1	275	278
6	2	18	2•
6	3	83	91
6	4	36	24
6	5	31	40
7	1	91	88
7	2	18	6•
7	3	26	12
7	4	40	37
8	1	26	16
8	2	16	0•
8	3	27	19
9	1	45	44
9	2	54	44
••• L = -16 •••			
1	0	550	563
1	1	163	163
1	2	212	216
1	3	214	220
1	4	158	152
1	5	37	25
1	6	43	38
2	0	30	25
2	1	84	80
2	2	24	11
2	3	39	25
2	4	128	122
2	5	44	40
2	6	14	13•
3	0	16	6•
3	1	122	120
3	2	65	62
3	3	28	28
3	4	18	2•
3	5	36	38
3	6	26	20
4	0	56	61
4	1	17	15•
4	2	128	123
4	3	18	21•
4	4	32	32
4	5	16	0•
4	6	42	43
5	0	100	98
5	1	52	44
5	2	142	148
5	3	18	3•
5	4	64	55
5	5	15	8•
6	0	27	20
6	1	118	121
6	2	32	38
6	3	39	35
6	4	43	43
6	5	74	79
7	0	103	108
7	1	95	93
7	2	46	47
7	3	60	68
7	4	65	70
8	0	54	65
8	1	64	61
8	2	26	28
8	3	25	19
8	4	35	35
9	0	59	60
9	1	15	0•
9	2	30	22
9	3	29	24
10	0	54	57
10	1	50	46
••• L = -15 •••			
1	1	246	242
1	2	142	139
1	3	329	338
1	4	164	163
1	5	112	113
1	6	63	62
2	1	85	77
2	2	189	187
2	3	27	19
2	4	252	256
2	5	18	14•
2	6	31	28
3	1	53	51
3	2	93	87
3	3	145	156
3	4	101	105
3	5	18	17•
3	6	36	36
4	1	17	8•
4	2	37	32
4	3	42	45
4	4	40	30
4	5	78	81
4	6	34	33
5	1	48	47
5	2	100	90
5	3	18	3•
5	4	105	104
5	5	43	37
6	1	226	231
6	2	91	87
6	3	125	125
6	4	42	47
6	5	40	36
7	1	56	53
7	2	18	8•
7	3	35	28
7	4	15	12•
8	1	27	17
8	2	17	11•
8	3	16	17•
8	4	32	29
9	1	37	25
9	2	40	40
9	3	29	35
10	1	13	11•
••• L = -14 •••			
1	0	146	148
1	1	63	64
1	2	165	176
1	3	145	143
1	4	29	20
1	5	18	13•
1	6	78	76
2	0	273	276
2	1	56	63
2	2	63	64
2	3	17	2•
2	4	28	2
2	5	49	43
2	6	96	84
3	0	112	124
3	1	74	86
3	2	53	48
3	3	60	58
3	4	27	20
3	5	18	4•
3	6	15	4•
4	0	381	386
4	1	83	87
4	2	86	88
4	3	18	10•
4	4	19	20•
4	5	91	87
4	6	14	1•
5	0	109	113
5	1	183	186
5	2	67	60
5	3	18	5•
5	4	18	17•
5	5	28	16
5	6	12	7•
6	0	530	547
6	1	79	75
6	2	77	76
6	3	74	83
6	4	77	80
6	5	15	5•
7	0	342	360
7	1	47	46
7	2	52	51
7	3	45	47
7	4	63	68
7	5	13	7•
8	0	84	77
8	1	69	66
8	2	17	0•
8	3	36	30
8	4	14	7•
9	0	34	25
9	1	26	21
9	2	15	5•
9	3	14	11•
10	0	35	36
10	1	44	34
10	2	68	63
••• L = -13 •••			
1	1	234	224
1	2	101	105
1	3	274	271
1	4	220	235
1	5	29	21
1	6	17	4•
2	1	344	330
2	2	80	78
2	3	25	34
2	4	127	123
2	5	51	54
2	6	73	71
3	1	28	18
3	2	111	110
3	3	112	109
3	4	145	143
3	5	78	67
3	6	27	27
4	1	131	138
4	2	104	95
4	3	94	90
4	4	39	42
4	5	18	18•
4	6	24	18
5	1	44	46
5	2	46	36
5	3	121	119
5	4	18	4•
5	5	17	12•
5	6	13	15•
6	1	218	224
6	2	123	118
6	3	27	20
6	4	52	53
6	5	37	30
7	1	194	198
7	2	85	89
7	3	31	33
7	4	16	25•
7	5	36	35
8	1	157	165
8	2	18	1•
8	3	17	11•
8	4	24	17
9	1	50	49
9	2	16	16•
9	3	14	4•
10	1	40	34
10	2	46	41
••• L = -12 •••			
1	0	152	143
1	1	129	122
1	2	79	72
1	3	35	28
1	4	41	42
1	5	42	37
1	6	54	50
1	7	27	20
2	0	422	410
2	1	250	238
2	2	102	96
2	3	70	48
2	4	470	483
2	5	80	76
2	6	70	68
3	0	42	36
3	1	186	179
3	2	119	118
3	3	24	7
3	4	147	155
3	5	58	60
3	6	26	17
4	0	133	132
4	1	107	108
4	2	202	210
4	3	69	71
4	4	168	169
4	5	46	47
4	6	28	21
5	0	194	199
5	1	48	52
5	2	17	1•
5	3	88	88
5	4	98	104
5	5	17	9•
5	6	14	3•
6	0	261	267
6	1	201	202
6	2	37	30
6	3	18	6•
6	4	110	116
6	5	16	8•
7	0	399	426
7	1	18	14•
7	2	27	14
7	3	18	20•
7	4	55	54
7	5	14	3•
8	0	18	20•
8	1	43	48
8	2	18	7•
8	3	109	114
8	4	15	6•
9	0	50	48
9	1	80	79
9	2	30	31
9	3	15	6•
9	4	34	25
10	0	15	22•
10	1	15	12•
10	2	48	41
••• L = -11 •••			
1	1	44	42
1	2	23	27
1	3	191	182
1	4	17	1•
1	5	49	45
1	6	85	81
1	7	67	67
2	1	204	199
2	2	49	47
2	3	202	205
2	4	107	101
2	5	56	68
2	6	75	72
2	7	39	38
3	1	219	222
3	2	189	179
3	3	53	47
3	4	123	129
3	5	19	3•
3	6	28	9
4	1	93	88
4	2	55	51
4	3	111	116
4	4	135	137
4	5	18	15•
4	6	31	23
5	1	55	55
5	2	55	49
5	3	33	34
5	4	58	59
5	5	61	60
5	6	14	3•
6	1	35	32
6	2	18	15•
6	3	18	4•
6	4	78	72
6	5	16	6•
6	6	23	15
7	1	224	226
7	2	41	35
7	3	99	105
7	4	17	15•
7	5	33	31
8	1	55	56
8	2	18	1•
8	3	76	79
8	4	44	44
9	1	17	8•
9	2	39	37
9	3	15	20•
9	4	42	39
10	1	62	53
10	2	24	22
10	3	25	23
••• L = -1 •••			
7	1	118	118
••• L = -10 •••			
1	0	176	179
1	1	30	18
1	2	237	232
1	3	15	10•
1	4	60	70
1	5	113	114
1	6	18	19•
1	7	41	39
2	0	636	635
2	1	606	601
2	2	28	6
2	3	380	380
2	4	74	77
2	5	91	91
2	6	89	84
2	7	13	6•
3	0	289	286
3	1	272	262
3	2	92	109
3	3	201	200
3	4	27	30
3	5	114	117
3	6	54	91
4	0	28	25
4	1	280	277
4	2	76	75
4	3	219	217
4	4	42	43
4	5	48	52
4	6	41	37
5	0	25	28
5	1	205	204
5	2	39	42
5	3	37	35
5	4	27	23
5	5	61	60
5	6	47	49
6	0	206	218
6	1	123	122
6	2	37	36
6	3	127	133
6	4	18	11•
6	5	42	44
6	6	13	7•
7	0	221	238
7	1	87	83
7	2	18	17•
7	3	105	102
7	4	37	42
7	5	36	24
8	0	342	357
8	1	41	34
8	2	91	89
8	3	41	39
8	4	70	71
8	5	31	27
9	0	85	89
9	1	18	9•
9	2	70	79
9	3	31	29
9	4	35	29
10	0	53	46
10	1	44	33
10	2	32	27
10	3	22	9
11	0	13	16•
11	1	12	12•
••• L = -9 •••			
1	1	172	180
1	2	276	262
1	3	111	110
1	4	33	37
1	5	127	130
1	6	161	149
1	7	24	7
2	1	272	268
2	2	79	73
2	3	52	54
2	4	133	137
2	5	42	40
2	6	48	47
2	7	14	5•
3	1	388	371
3	2	104	90
3	3	85	81
3	4	45	44
3	5	286	290
3	6	120	118
3	7	23	12
4	1	118	123
4	2	42	52
4	3	101	110
4	4	18	18•
4	5	142	143
4	6	61	68
5	1	79	73
5	2	194	196
5	3	43	33
5	4	33	20
5	5	18	16•
5	6	25	15
6	1	60	60
6	2	65	61
6	3	104	101
6	4	46	44
6	5	17	19•
6	6	13	14•
7	1	144	145
7	2	18	11•
7	3	18	37•
7	4	18	7•
7	5	28	18
8	1	151	154
8	2	46	42
8	3	36	38
8	4	36	23
8	5	22	17
9	1	42	39
9	2	17	25•
9	3	36	34
9	4	29	28
10	1	16	4•
11	1	13	13•
10	2	15	20•
10	3	25	18
••• L = -8 •••			
1	0	582	575
1	1	231	222
1	2	100	102
1	3	197	190
1	4	24	11
1	5	18	25•
1	6	83	78
1	7	38	43
2	8	525	534
2	1	191	194
2	2	441	442
2	3	126	125
2	4	95	91
2	5	139	146
2	6	118	113
2	7	14	8•
3	0	128	130
3	1	275	268
3	2	345	337
3	3	58	56
3	4	168	171
3	5	139	134
3	6	98	100
3	7	13	2•
4	0	107	103
4	1	70	64
4	2	185	183
4	3	128	126
4	4	18	11•
4	5	73	70
4	6	57	51
5	0	92	101
5	1	49	49
5	2	226	225
5	3	47	41
5	4	46	46
5	5	18	7•
5	6	54	51
6	0	65	69
6	1	197	199
6	2	43	38
6	3	18	4•
6	4	37	34
6	5	17	1•
6	6	28	20
7	0	249	260
7	1	47	48
7	2	18	2•
7	3	18	22•
7	4	18	10•
7	5	46	34
8	0	421	437
8	2	113	114
8	3	95	103
8	1	103	96
8	4	78	82
8	5	26	18
9	0	151	157
9	1	86	90
9	2	18	6•
9	3	16	6•
9	4	26	29
10	0	63	59
10	1	16	0•
10	2	33	34
10	3	46	42
11	0	66	61
11	1	29	31
••• L = -7 •••			
1	1	309	303
1	2	17	12
1	3	127	115
1	4	69	76
1	5	56	56
1	6	50	48
1	7	15	18•
2	1	293	290
2	2	308	314
2	3	126	124
2	4	17	10•
2	5	30	20
2	6	46	42
2	7	15	5•
3	1	38	41
3	2	693	685
3	3	82	87
3	4	196	194
3	5	151	156
3	6	78	74
3	7	14	1•
4	1	214	217
4	2	288	273
4	3	30	30
4	4	30	37
4	5	29	18
4	6	26	2
5	1	139	142
5	2	115	104
5	3	110	104
5	4	34	29
5	5	88	86
5	6	31	25
6	1	149	143
6	2	122	118
6	3	18	3•
6	4	70	75
6	5	17	1•
6	6	38	36
7	1	149	149
7	2	34	30
7	3	59	66
7	4	18	16•
7	5	36	32
8	1	70	73
8	2	73	67
8	3	109	113
8	4	64	60
8	5	51	48
9	1	74	74
9	2	74	74
9	3	47	43
9	4	38	32
10	1	16	7•
10	2	78	73
10	3	73	69
11	1	35	30
11	2	12	3•
••• L = -6 •••			
1	0	1233	1253
1	1	203	202
1	2	350	356
1	3	81	73
1	4	141	135
1	5	74	77
1	6	18	1•
1	7	15	12•
2	0	138	132
2	1	41	36
2	2	408	401
2	3	120	120
2	4	44	38
2	5	93	98
2	6	18	3•
2	7	51	46
3	0	375	387
3	1	607	601
3	2	285	285
3	3	76	75
3	4	45	46
3	5	55	56
3	6	18	4•
3	7	31	25
4	0	249	251
4	1	13	6•
4	2	424	417
4	3	132	137
4	4	25	9
4	5	138	140
4	6	43	33
4	7	26	25
5	0	33	15
5	1	333	330
5	2	23	11
5	3	17	20•
5	4	65	62
5	5	94	97
5	6	56	54
6	0	141	153
6	1	62	70
6	2	102	100
6	3	167	164
6	4	74	74
6	5	41	31
6	6	40	39
7	0	31	27
7	1	92	90
7	2	109	114
7	3	82	81
7	4	35	30
7	5	33	30
8	0	110	115
8	1	153	160
8	2	30	26
8	3	101	103
8	4	17	12•
8	5	68	65

Table 4 (cont.)

The table below is a list of observed and calculated structure factors, arranged in repeating column groups of **H K FOBS FCAL**. The groups are transcribed left-to-right. An asterisk (*) marks values as printed.

```
H  K  FOBS FCAL      H  K FOBS FCAL      H  K FOBS FCAL      H  K FOBS FCAL
9  0  38   32        8  3  34   32       8  0 187  195       7  2 112  113
9  1  36   31        8  4  17   18*      8  1  71   73       7  3  76   79
9  2  89    9*       8  5  35   21       7  4 149  151       7  4  30   15
9  3  17    3*       9  0  77   81       7  5  17    5*      7  5  17    5*
9  4  35   27        9  1  95   89       7  6  24   20       7  6  24   20
10 0  17   14*       9  2  73   69       8  0  94  101       8  0  94  101
10 1  33   26*       9  3  17   11*      8  1 111  115       8  1 111  115
10 2  80   87        9  4  77   71       8  2  80   80       8  2  80   80
10 3  28   22       10  0  65   59       8  3  27   18       8  3  27   18
11 0  14    2*      10  1  14    2*      8  4  17    7*      8  4  17    7*
11 1  14    9*      10  2  31   28       8  5  97   84       8  5  97   84
11 2  25   25       10  3  14    2*      9  0  18   12*      9  0  18   12*
*** L = -5 ***      11  0  14    9*      9  1  68   67       9  1  68   67
1  1 374  370       11  1  42   35       9  2 213  219       9  2 213  219
1  2 186  183       11  2  13    1*      9  3  63   68       9  3  63   68
1  3  38   29       *** L = -3 ***       9  4  39   34       9  4  39   34
1  4 146  142        1  1 910  938      10  0  17    8*     10  0  17    8*
1  5 103  103        1  2 350  358      10  2 114  110      10  2 114  110
1  6  18   15*       1  3 114  113      10  3  59   55      10  3  59   55
1  7  26   17        1  4  32   17      11  0  15    7*     11  0  15    7*
2  1 245  243        1  5  27   29      11  1  40   36      11  1  40   36
2  2 260  262        2  1  19    4*     11  2  33   28      11  2  33   28
2  3  39   41        2  2 845  839      *** L = 1 ***       *** L = 1 ***
2  4 113  113        2  3  20   12       0  1 752  777       0  1 752  777
2  5  18    6*       2  4 115  123       0  2  70   69       0  2  70   69
2  6  92  100        2  5  71   30       0  3 182  179       0  3 182  179
2  7  15    3*       2  6  18   16*      0  4  47   37       0  4  47   37
3  1  78   84        2  7  15    7*      0  5  34   24       0  5  34   24
3  2 517  510        3  1 390  380       0  6  43   43       0  6  43   43
3  3  15    3*       3  2 106   98       1  1 320  315       1  1 320  315
3  4  91   94        3  3  45   41       1  2 262  269       1  2 262  269
3  5 241  245        3  4  45   41       1  3 155  159       1  3 155  159
3  6 123  126        3  5 100  107       1  4 233  241       1  4 233  241
3  7  27   20        3  6  13   65       1  5 112  110       1  5 112  110
4  1 487  481        3  7  15    6*      1  6  46   43       1  6  46   43
4  2 228  220        4  1 105  102       1  7  59   62       1  7  59   62
4  3 213  220        4  2 385  372       2  1 114  112       2  1 114  112
4  4  91   92        4  3 187  191       2  2 144  150       2  2 144  150
4  5 250  252        4  4  42   35       2  3  16    6*      2  3  16    6*
4  6  28   23        4  5 209  214       2  4 184  189       2  4 184  189
4  7  13   22*       4  6  18   17*      2  5  52   47       2  5  52   47
5  1  45   46        4  7  13   11*      2  6  19   42*      2  6  19   42*
5  2 367  368        5  1 122  120       3  1  81   73       3  1  81   73
5  3  88   91        5  2 131  132       3  2  40   39       3  2  40   39
5  4  18    4*       5  3  37   32       3  3  84   87       3  3  84   87
5  5  30   16*       5  4  72   71       3  4  38   37       3  4  38   37
5  6  68   62        5  5  18    3*      3  5  65   70       3  5  65   70
6  1  16   11*       5  6  80   79       3  6  37   28       3  6  37   28
6  2  46   36        6  1  62   68       4  1 220  216       4  1 220  216
6  3  81   81        6  2 161   96       4  2  20   18       4  2  20   18
6  4  13   20*       6  3  18   13*      4  3  96  100       4  3  96  100
6  5  43   42        6  4  56   95       4  4 211  215       4  4 211  215
6  6  15    2*       6  5  31   28       4  5  87   63       4  5  87   63
7  1 198  205        6  6  58   59       5  0 105  107       5  0 105  107
7  2  18    2*       7  1  31   23       5  1  33   28       5  1  33   28
7  3  27   18        7  2  31   21       5  2 465  445       5  2 465  445
7  4  33   29        7  3  70   68       5  3 226  232       5  3 226  232
7  5  16   12*       7  4  98   98       5  4  41   43       5  4  41   43
7  6  12    8*       7  5  17   13*      5  5 213  226       5  5 213  226
8  1 200  205        7  6  44   48       5  6  17    4*      5  6  17    4*
8  2  53   47        8  1  29   21       6  1  25   16       6  1  25   16
8  3  38   41        8  2  33   21       6  2  49   53       6  2  49   53
8  4  17    5*       8  3  45   40       6  3 137  145       6  3 137  145
8  5  14   15*       8  4  17   19*      6  4  18   14*      6  4  18   14*
9  1 284  285        8  5  27   14*      6  5  35   33       6  5  35   33
9  2  18   11*       9  1 111  106       7  2  79   87       7  2  79   87
9  3 103  104        9  2  18   10*      7  3  53   55       7  3  53   55
9  4  15   10*       9  3  31   20       7  4  63   64       7  4  63   64
10 1  70   69        9  4  15    7*      7  5  42   45       7  5  42   45
10 2  16   11*      10  1  38   33       7  6  40   38       7  6  40   38
10 3  14    8*      10  2  54   51       8  1  39   40       8  1  39   40
11 2  24   20       10  3  15   15*      8  2  18    8*      8  2  18    8*
*** L = -4 ***      11  1  25   28       8  3  35   30       8  3  35   30
1  0 400  408       11  2  13    9*      8  4  17   15*      8  4  17   15*
1  1 298  296       *** L = -2 ***       9  2 108  109       9  2 108  109
1  2 162  170        1  0 180  178       9  3  64   60       9  3  64   60
1  3  22   23        1  1  41   38       9  4  30   24       9  4  30   24
1  4 137  139        1  2  45   37      10  1  70   70      10  1  70   70
1  5  34   30        1  3  30   25      10  2 105  100      10  2 105  100
1  6  32   35        1  4  66   67      10  3  26   25      10  3  26   25
1  7  35   33        1  5 475  484      11  1  59   57      11  1  59   57
2  0 730  721        1  6  19   12*     11  2  79   79      11  2  79   79
2  1 429  424        1  7  29   24      *** L = 2 ***       *** L = 2 ***
2  2 427  431        2  0 760  756       0  0 623  649       0  0 623  649
2  3  13    6*       2  1 235  237       0  1 135  128       0  1 135  128
2  4 108  113        2  2 432  441       0  2 236  243       0  2 236  243
2  5  50   45        2  3 130  133       0  3  27   10       0  3  27   10
2  6 143  146        2  4 179  191       0  4  57   52       0  4  57   52
2  7  15   11*       2  5  37   35       0  5  19    7*      0  5  19    7*
3  0  35   32        2  6  39   33       0  6 365  364       0  6 365  364
3  1  60   51        2  7 184  182       1  0  99   92       1  0  99   92
3  2 243  247        3  0  71   66       1  1  24   25       1  1  24   25
3  3  75   77        3  1  26   24       1  2  47   58       1  2  47   58
3  4 114  117        3  2 177  187       1  3 150  154       1  3 150  154
3  5 169  162        3  3  18   21*      1  4  44   39       1  4  44   39
3  6  67   59        4  0 438  432       1  5  40   34       1  5  40   34
3  7  14    8*       4  1 527  512       1  6  19   12*      1  6  19   12*
4  0 586  577        4  2  54   48       1  7  16   25*      1  7  16   25*
4  1 742  728        4  3 220  219       2  0 572  557       2  0 572  557
4  2  57   43        4  4 216  224       2  1 399  393       2  1 399  393
4  3  76   68        4  5 183  187       2  2 199  204       2  2 199  204
4  4 102  102        4  6  30   25       2  3  78   71       2  3  78   71
4  5 225  233        5  0  24   33       2  4 168  170       2  4 168  170
4  6 112  114        5  1 290  277       2  5  18   24*      2  5  18   24*
4  7  13   19*       5  2 106   98       2  6  15   16*      2  6  15   16*
5  0  68   69        5  3  93   80       2  7  30   32       2  7  30   32
5  1 130  118        5  4 235  264       3  0 312  305       3  0 312  305
5  2 212  209        5  5  78   74       3  1 312  305       3  1 312  305
5  3  17   10*       5  6  39   37       3  2 111   98       3  2 111   98
5  4 115  120        5  7  18    8*      3  3  40   33       3  3  40   33
5  5  18    4*       6  0 151  151       3  4  29   29       3  4  29   29
5  6  80   73        6  1  60   54       3  5  40   43       3  5  40   43
6  0  63   58        6  2  18   19*      3  6  16   14*      3  6  16   14*
6  1 100   90        6  3  80   86       3  7  16    4*      3  7  16    4*
6  2 105  100        6  4  51   43
6  3  54   56        6  5 235  240
6  4  38   42        6  6  81   84
6  5  68   67        7  0  29   29
6  6  15   16*       7  1 120  110
7  0  44   38        7  2  85   83
7  1  17    5*       7  3  69   68
7  2  18   16*       7  4  71   70
7  3  18   10*       7  5  33   26
7  4  69   62        7  6  15   16*
7  5  49   42        7  7  16   21*
7  6  12   13*
8  0  71   70
8  1  94   90
8  2 113  118
```

Table 4 (cont.)

The columns are repeated across the page with headers: H K FOBS FCAL

Table 5. Bond distances involving hydrogen atoms

The estimated standard deviations are 0·02 Å.

C(2)—H(24)	0·99 Å
C(2)—H(25)	0·95
C(3)—H(26)	0·98
C(3)—H(27)	1·00
C(4)—H(28)	1·02
C(4)—H(29)	0·96
C(5)—H(30)	0·98
C(5)—H(31)	1·02
C(6)—H(32)	1·02
C(7)—H(33)	1·04
C(7)—H(34)	1·03
C(8)—H(35)	0·94
C(8)—H(36)	1·00
C(11)—H(37)	0·95
C(13)—H(38)	0·89
C(15)—H(39)	0·94

Table 6. Bond angles involving hydrogen atoms

The estimated standard deviations are in 1° for angles involving only one hydrogen atom and 2° for angles involving two hydrogen atoms.

H(24)-C(2)—C(1)	111°
H(24)-C(2)—C(3)	114
H(25)-C(2)—C(1)	103
H(25)-C(2)—C(3)	111
H(26)-C(3)—C(2)	113
H(26)-C(3)—C(4)	107
H(27)-C(3)—C(2)	114
H(27)-C(3)—C(4)	107
H(28)-C(4)—C(3)	108
H(28)-C(4)—C(5)	112
H(29)-C(4)—C(3)	107
H(29)-C(4)—C(5)	111
H(30)-C(5)—C(4)	113
H(30)-C(5)—C(6)	111

Table 6 (*cont.*)

H(31)–C(5)—C(4)	113
H(31)–C(5)—C(6)	108
H(32)–C(6)—C(1)	102
H(32)–C(6)—C(5)	106
H(32)–C(6)—C(7)	108
H(33)–C(7)—C(6)	113
H(33)–C(7)—C(8)	111
H(34)–C(7)—C(6)	122
H(34)–C(7)—C(8)	118
H(35)–C(8)—C(1)	110
H(35)–C(8)—C(7)	113
H(36)–C(8)—C(1)	120
H(36)–C(8)—C(7)	122
H(37)–C(11)–C(10)	122
H(37)–C(11)–C(12)	118
H(38)–C(13)–C(12)	120
H(38)–C(13)–C(14)	123
H(39)–C(15)–C(10)	119
H(39)–C(15)–C(14)	122
H(24)–C(2)—H(25)	109
H(26)–C(3)—H(27)	98
H(28)–C(4)—H(29)	104
H(30)–C(5)—H(31)	106
H(33)–C(7)—H(34)	106
H(35)–C(8)—H(36)	104

A comparison of σ, the estimated standard deviations calculated from the least-squares matrices, and σ_a, the root mean square deviation from the averages of some sets of chemically similar bonds, is shown in Table 7. This comparison suggests that the deviations calculated from the least-squares matrices are underestimates, as is usually the case. The true uncertainties are thus higher, but by an amount difficult to assess, than those shown in the Tables.

Table 7. *Comparison of standard deviations*

Values of σ were obtained from the least-squares matrices; values of σ_a are root mean square deviations from the averages of chemically similar bonds.

Bond type	σ	σ_a
C–C (benzene)	0·003 Å	0·005 Å
N–O	0·003	0·005
C–H	0·02	0·04

The bond distances in the four-membered ring of *trans*(4.2.0) are very close to that reported for cyclobutane (1·548 Å; Almenningen, Bastiansen & Skancke, 1961). The average C–C bond distance (1·544 Å) in the cyclobutane ring of *trans*(4.2.0) is slightly longer than that reported for n-butane (1·533 Å; Bonham & Bartell, 1959). The elongation of the C–C bonds in this cyclobutane ring is similar to that observed for cyclobutane rings which are not part of condensed polycyclic systems. The average bond angle in the cyclobutane ring of *trans*(4.2.0), 88·0°, is not significantly different from that reported for cyclobutane (Skancke, 1960). In the substituted cyclobutanes which are not planar, the dihedral angle in the ring varies from 160° in octafluorocyclobutane (Lemoire & Livingston, 1952) to 145° in cyclobutane. (For a summary of dimensions of

cyclobutane rings, see Adman & Margulis, 1968.) This dihedral angle in *trans*(4.2.0) is 147°. The close similarity of ring bond distances and angles and ring conformation of the cyclobutane ring in *trans*(4.2.0) and in non-fused systems suggests that little or no additional strain is introduced into this ring by *trans* fusion with a cyclohexane ring.

Ring conformations may be conveniently described in terms of an angle of twist about each ring bond (Brown & Levy, 1965). Values of these conformational angles for the bicyclic system of *trans*(4.2.0) are shown in Table 8. The calculation of C(6) → C(1) includes adjacent atoms in the six-membered ring, while that denoted as C(1) → C(6) refers to the four-membered ring. Fig. 3 is a stereoscopic view of the bicyclic system in which the ester oxygen atom and the hydrogen atoms are included so that the *trans* configuration and the ring conformations can be more easily seen.

Table 8. *Conformational angles of directed bonds in the bicyclic system*

	Angle
C(1) → C(2)	−54·3°
C(2) → C(3)	49·7
C(3) → C(4)	−50·9
C(4) → C(5)	51·0
C(5) → C(6)	−56·8
C(6) → C(1)	62·9
C(6) → C(7)	23·3
C(7) → C(8)	−22·9
C(8) → C(1)	23·0
C(1) → C(6)	−23·1

The six-membered ring of the bicyclic system is in the chair conformation. The bond distances adjacent to the fused bond are significantly shorter than the usual C–C bond. The chair conformation of the cyclohexane ring is considerably flattened; this can be seen by a comparison of the conformational angles in this ring with the ideal value of 60° for chair cyclohexane. It appears, then, that the ring strain in this bicyclic system is almost entirely accommodated by the more flexible six-membered ring, leaving the cyclobutane ring essentially undistorted.

Bond distances and angles in the dinitrobenzoate group appear to be normal. The angles in the phenyl ring at the carbon atoms attached to the nitro groups are both greater than 120°, a common occurrence in nitrobenzenes (Carter, McPhail & Sim, 1966). The planes of the nitro groups make angles 4·3° and 1·4° with the plane of the phenyl ring. The group including N(22) – that is, the one nearest the C=O bond of the carboxylate group – is the one which is twisted most out of the phenyl plane. An even larger angle, 8·9°, is made by the carboxylate plane and the phenyl plane, but these angles are all relatively small, and the dinitrobenzoate group can be considered to be approximately planar.

One outstanding gross feature of the structure which has already been mentioned is the approximate planarity

of a large portion of the molecule. The least-squares plane $0.5030X + 0.4808Y + 0.7182Z = 5.934$, calculated including the dinitrobenzoate group and the bridgehead carbon atoms (17 atoms out of 23) has only three atoms with deviation of 0.2 Å from the plane, but no others more than 0.1 Å from the plane; the coefficients in the above equation are the direction cosines of the normal to the plane relative to **a**, **b** and **c***.

The structure contains no unusually short intermolecular contact distances. Fig. 4 shows a stereoscopic drawing of a packing diagram. The direction of view is approximately along the y axis, with the x axis across the Figure. The box indicates the volume of one unit cell $(0 \leq x \leq a, \ 0 \leq y \leq b, \ 0 \leq z \leq c)$; the sixteen molecules which extend into this unit cell are shown.

References

ADMAN, E. & MARGULIS, T. N. (1968). *J. Amer. Chem. Soc.* **90**, 4517.
ALMENNINGEN, A., BASTIANSEN, O. & SKANCKE, P. N. (1961). *Acta Chem. Scand.* **15**, 711.
BEDNOWITZ, A. (1966). Private communication.
BONHAM, R. A. & BARTELL, L. S. (1959). *J. Amer. Chem. Soc.* **81**, 3491.
BROWN, G. M. & LEVY, H. A. (1965). *Science*, **141**, 921.
CARTER, O. L., MCPHAIL, A. T. & SIM, G. A. (1966). *J. Chem. Soc.* p. 822.
CROMER, D. T. & WABER, J. T. (1965). *Acta Cryst.* **18**, 104.
EVANS, H. T. (1961). *Acta Cryst.* **14**, 689.
GVILDYS, J. (1965). Argonne National Laboratory Program B-149.
JOHNSON, C. K. (1965). Program *ORTEP*, revised. ORNL-3794, Oak Ridge National Laboratory, Oak Ridge, Tennessee.
KARLE, J. & KARLE, I. L. (1966). *Acta Cryst.* **21**, 849.
KARTHA, G. (1964). Private communication.
LEMOIRE, H. P. & LIVINGSTON, R. L. (1952). *J. Amer. Chem. Soc.* **74**, 5732.
MEINWALD, J., ANDERSON, P. & TUFARIELLO, J. (1967). *J. Amer. Chem. Soc.* **88**, 1301.
SKANCKE, P. N. (1960). Thesis, Oslo. Referenced by Wiberg & Hess (1967).
STEWART, R. F., DAVIDSON, E. R. & SIMPSON, W. T. (1965). *J. Chem. Phys.* **42**, 3175.
TULINSKY, A., WORTHINGTON, C. R. & PIGNATARO, E. (1959). *Acta Cryst.* **12**, 623.
WIBERG, K. B. & HESS, B. A. JR (1967). *J. Amer. Chem. Soc.* **89**, 3015.
WIBERG, K. B. (1967). Private communication.
WILLIAMS, J. M. (1966). Argonne National Laboratory Program MET176X.

The Crystal Structure of Zinc Guanidinium Sulfate

BY CARL N. MORIMOTO AND E. C. LINGAFELTER

Department of Chemistry, University of Washington, Seattle, Washington 98105, *U.S.A.*

The crystal structure of zinc guanidinium sulfate, $Zn[C(NH_2)_3]_2(SO_4)_2$, has been determined by a three-dimensional X-ray analysis. The crystals are tetragonal, space group $I\bar{4}2d$, with cell dimensions of $a = 9.515$ and $c = 14.351$ Å. There are four formula units in the unit cell, with density calculated to be 1.929 g.cm^{-3} in agreement with the measured density (1.93 g.cm^{-3}). The structure has been determined by Patterson and Fourier methods from 741 reflections collected by counter and refined by least-squares to a conventional R index of 0.017. A network of zinc and sulfate ions constitutes the main feature of the structure; the sulfate ions are arranged tetrahedrally around the zinc ions with the Zn–O distance being 1.94 Å. The sulfate ion, on a twofold axis, has one pair of oxygen atoms coordinated to zinc ions, resulting in an S–O bond distance of 1.49 Å, which is significantly longer than the S–O distance of 1.44 Å for the second pair of sulfate oxygen atoms.

Introduction

The synthesis of a series of hexahydrated double salts between guanidinium sulfate and bivalent metal sulfates was reported by Canneri (1925). It was of interest to investigate the metal ion coordination in the bivalent metal guanidinium sulfate hexahydrates (M(II)GSH from here on), $M(II)[C(NH_2)_3]_2 (SO_4)_2 . 6H_2O$, for comparison with the ammonium Tutton salts, $M(II)(NH_4)_2 (SO_4)_2 . 6H_2O$, which have been studied extensively (see Montgomery, Chastain, Natt, Witkowska & Lingafelter, 1967). In an attempt to prepare ZnGSH by room temperature evaporation of the aqueous solution of the mixed salts, crystals were obtained which appeared to be monoclinic as reported for M(II)GSH (Canneri, 1925). Although there was no evidence to indicate that these crystals were the desired complex, the structure determination by X-ray diffraction was undertaken. Later the crystal was identified as tetragonal and anhydrous instead of monoclinic and hexahydrate. Ef-